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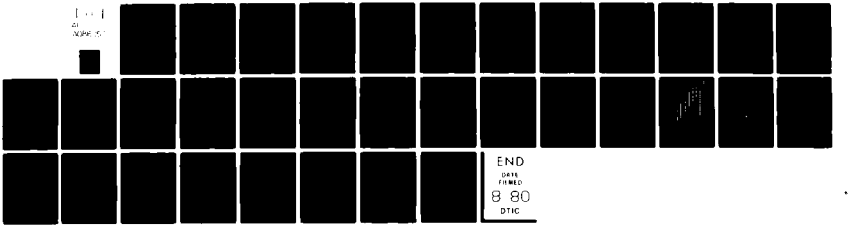

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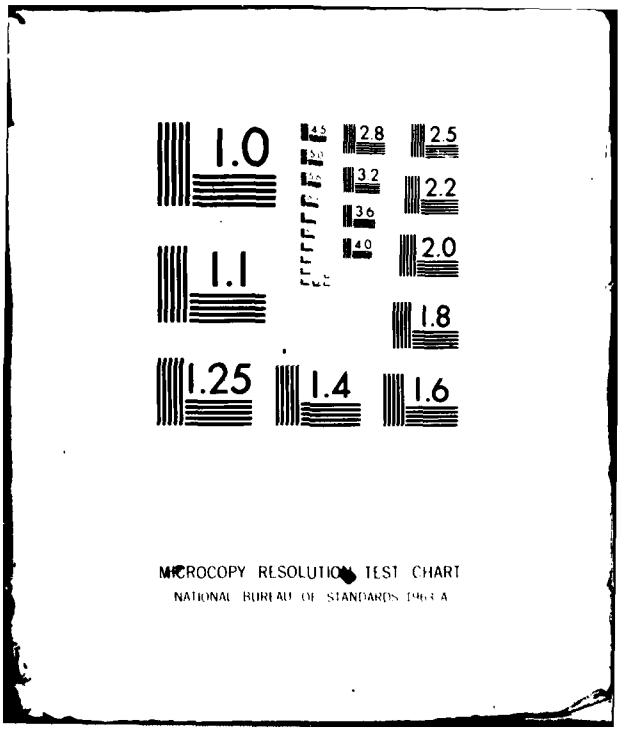
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MELT PURIFICATION VIA FILTRATION

April 1980

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Prepared for

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The apparatus under construction for steel filtration is described and the envisaged method of experimentation is discussed. Reference is made to proposed future work on a low temperature model system and to further studies of aluminium filtration.

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The apparatus under construction for steel filtration is described and the envisaged method of experimentation discussed. Reference is made to proposed future work on a low temperature model system and to further studies of aluminum filtration.

FOREWARD

This report was prepared by Drexel University summarizing the progress during the first six months on the feasibility of inclusion removal from molten metal systems. The contract, DAAG46-79-C0052 is being administered under the direction of Mr. Arthur Ayvazian of the U.S. Army Materials and Mechanics Research Center, Watertown, Massachusetts.

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1. INTRODUCTION

It is most desirable to remove impurities from the melt before casting. Technologically, removal of impurities improves the properties of the cast product, i.e., increased ductility and improved fatigue life. Financially, presence of impurities in the melt may cause production impedances and down time. Removal of non-metallic inclusions, trapped oxides of the metal, and extraneous particles can be accomplished by filtration - by passing the liquid metal through a packed bed of refractory particles,[1] screens,[2] ceramic foam filters[3] and subjecting the melt to various other purification methods.[4,5] Filtration of aluminum and its alloys, for instance, has proven to have beneficial effects on the physical and surface properties of the product.[3,6]

The impurities which are being filtered out may be characterized as:

(i) exogeneous inclusions, intermetallic compounds that nucleate and are suspended within the melt such as Al_2O_3 and SiO_2 deoxidation products in steel.

(ii) oxide of the melt which is both suspended on top of the melt and is entrapped within it due to turbulence - e.g. Al_2O_3 in aluminum melts.

The impurities in question are in the size range of 1-30μm, diameter.

Removal of second phase particles of the same size range, 1-30μm, from aqueous and organic solutions by filtration has been extensively studied; however, for the case of liquid metals (ferrous and non-ferrous) a thorough systematic study of filtration does not exist. This is perhaps due to the experimental difficulties involved (high temperatures) as well as the inherent constraints of the process.

The object of the proposed research program is to study and evaluate the feasibility of purifying aluminum, steel and nickel-based superalloys by filtration and to evaluate the characteristics of the filtered metals.

2. BACKGROUND

Refining of liquid metals requires the removal of inclusions in the size range of 1-30μm which are dilutely suspended (about 0.03% in steel). Furthermore, the filtration operating pressure should be moderate to make it economically justifiable.

There are two well known mechanisms of filtration: (i) surface filtration, and (ii) deep bed filtration. Surface filtration is normally used for high-solid suspensions while deep bed filters are used for dilute ones (0.01 to 0.05 vol %). A depth filter disperses the inclusions through part or all of its volume (depth). In surface filtration one relies on cake formation for particle retention and hence the size of the filter pores must be smaller than that of the impurities. The latter would require high operating pressures and thus surface filtration is impractical for most metallurgical applications.

2.1 MECHANISMS AND KINETICS OF FILTRATION

When a suspension of small particles flows past the grains of a filter medium, the particles deposit on the surface of the grain, due to diffusion, direct interception, gravity, and/or surface forces. These attachment forces can be classified to be either mechanical, physiochemical or a combination of both. Mechanical entrapment has been observed to be responsible for filtration of large particles (over 30μm) where volume phenomena prevail. In physiochemical mechanisms, surface forces are the dominant attachment forces for filtration of small particles in the range of 1μm. For this mode, it is

believed that the primary mode of transport of impurity particles to the grain surface of the filter medium is due to flow dynamics, while surface forces are responsible for retention of the particle to the grain surface.

In order to bridge the gap between the microscopic entrapment mechanisms and macroscopic filtration properties, a kinetic equation for filtration at a given liquid velocity has been used having the form[7,8]:

$$\left(\frac{\partial \sigma}{\partial t}\right)_Z = KC \qquad (1)$$

where

- σ is the volume of entrapped impurity particles per unit filter volume
- t is time
- K is the kinetic parameter
- C is the concentration of suspended impurity particles in the bulk liquid
- Z is the distance from the inlet of the filter

The above expression represents the rate of change of entrapped particles per unit filter volume. The parameter K has been found in low temperature systems to be a function of the volume of particles already retained in the filter bed, σ, and is expressed as:

$$K = K_o F(\sigma) \qquad (2)$$

where K_o is a constant whose value depends upon the properties of the fluid, as well as the size and morphology of both the filter medium and the impurity particles.

A great deal of work in depth filtration of low temperature systems - mostly aqueous - has been carried out over the years and many forms of the kinetic constant, K, have been proposed. Various published forms of the

kinetic constant for filtration in aqueous systems have been reported[9-19]. Work to date on the relevant capture processes (low temperature systems) sheds some light on the mechanisms of filtration; unfortunately, these are not applicable to liquid metals, since distinct differences exist. In liquid metals, temperatures are elevated, thus diffusional forces become important, and since the fluid is electrically conductive the electrokinetic forces do not play a dominant role.

2.2 PROPOSED KINETIC MODEL

Consider a differential section of the filter bed of cross-sectional area A and thickness ΔZ at a distance Z from the inlet, as shown in Figure 1.

$$\frac{\partial}{\partial t}\ (\sigma + \varepsilon C)\ + U_m \frac{\partial C}{\partial Z} = 0 \qquad (3)$$

In the above mass balance ε is the porosity of the bed, C is the volumetric concentration of inclusions in the melt, σ is the volume of entrapped inclusions per unit volume of filter grain and U_m is the superficial melt velocity. Solving the above with the appropriate boundary conditions:

$$\sigma = 0,\ \tau = 0 \text{ for all } Z$$

$$C = C_i, Z = 0 \text{ for all } \tau$$

(where C_i is the initial concentration of inclusions in the melt, and τ is the characteristic time[20] gives the filtered melt concentration, C:

$$C = C_i \frac{\exp\left[\frac{C_i}{\sigma_m} \cdot \varepsilon_o \cdot \phi(\theta-\eta)\right]}{\exp\ (\phi\eta) + \exp\left[\frac{C_i}{\sigma_m} \cdot \varepsilon_o \cdot \phi(\theta-\eta)\right] - 1} \qquad (4)$$

where the dimensionless parameters are defined as:

$\theta = \frac{tU_m}{\varepsilon_o L}$, dimensionless time; ε_o is the initial filter porosity and L is the filter length

$\eta = \frac{Z}{L}$, dimensionless distance

$\phi = K_o \frac{L}{U_m}$, dimensionless kinetic parameter

During short times, for both the variable kinetic parameter and constant kinetic parameter cases, the outlet concentration, Co, has the same functionality. In the variable kinetic parameter case:

$$K(\sigma) = K_o \left(1 - \frac{\sigma}{\sigma_m}\right) \tag{5}$$

where K_o is a constant and σ_m is the maximum entrapped inclusion concentration expressed as inclusion volume per filter volume; here when σ reaches σ_m filtration ceases - $K(\sigma) = 0$. In the case where the kinetic parameter is a constant:

$$K = K_o \tag{6}$$

here the rate of inclusion deposition on the filter grain surface is independent of the entrapped inclusion concentration. The expression for the melt outlet concentration (C_o) for short times (initial life of the filter) is:

$$C_o = C_i \exp\left(-\frac{K_o}{U_m} L\right) \tag{7}$$

Since K_o may be effected by the melt velocity U_m, we lump these two parameters together and represent their ratio $\frac{K_o}{U_m}$ by λ which is termed the filtration coefficient.

$$C_o = C_i e^{-\lambda L} \tag{8}$$

Thus to obtain good filtration efficiencies, one should either increase L or increase λ. The value of λ is of paramount importance in evaluating the feasibility of filtration processes.

Even though the parameter K_o is the fundamental parameter describing the capture rate during filtration, the λ term is more useful in the sense that it is directly related to the removal efficiency given by:

$$\text{efficiency} = \frac{C_i - C_o}{C_i} = 1 - e^{-\lambda L} \qquad (9)$$

One can increase the removal efficiency by either using a long filter (increase L), or operate a filter of given L at lower velocities or increasing K_o (increase λ).

Let us consider a specific length of the filter L such that the inclusion removal efficiency is 63.2%. In other words, consider the value of L so that $(1 - e^{-\lambda L}) = 0.632$. The latter is only satisfied when L equals $(1/\lambda)$ or (U_m/K_o). We will now define the parameter Λ which is equal to U_m/K_o and has the units of distance, cm. The physical significance of Λ is that it is the depth of the filter needed for 63.2% removal of incoming inclusions. Λ can be thought of as a unit removal length - a high Λ value indicating a "poor" filter, or that a deeper filter is required for a 63.2% removal.

3. RESEARCH OBJECTIVES

The goal of the proposed program is to study and evaluate the feasibility and utility of purifying liquid metals by filtering it prior to casting. More specifically, the various tasks that need to be investigated are:

- evaluate various filter media in terms of serviceability and compatibility.
- model the filtration process for liquid metals to quantify the particle capture kinetics.
- develop the required process parameters, i.e., approach velocity of the melt, porosity of the filter bed and pressure drop characteristics.
- optimum design of the filter for specific alloy systems such as aluminum, deoxidized steel, and superalloys.
- evaluate the mechanical properties of the filtered metal - reduction of area, tensile, impact and fatigue properties.

The program schedule is shown in Figure 2.

4. PROGRESS TO DATE

In order to gain experience with the design of apparatus and the required experimental techniques, the work to date has concentrated on filtering molten aluminum. As the technology necessary for the filtration of aluminum has now been developed, this expertise is currently being applied to the design and construction of an apparatus for steel filtration, so that the next phase of the work may be initiated. Also in order to obtain a more complete characterization of the mechanisms involved in the filtration of molten metals, modeling of the filtration process via a low temperature system is being pursued. The latter is an added task, however it is felt that data obtained through the model system will be extremely valuable. Each of these fields of activity is further discussed below.

4.1 Experimental Design and Procedure for Aluminum Filtration

An experimental apparatus was designed which took into account the corrosive nature of molten aluminum, the requirements of sustained heating, adequate filter depth and metal flowrates in the range of 1-17 $Kg/(m^2 \cdot s)$ (filter factor of 5-90 $lbs/in^2 \cdot hr$). The metal throughput per filter area is of great importance for commercial applications and hence the experiments were designed to simulate industrial filter factor levels.

The "deep bed" filter apparatus consists of a 6 in x 40 in SiC tube placed in a resistance furnace (Figure 3). The orifice size at the bottom of the tube and the metallostatic head above it control the metal flowrate. The filter medium consists of a 46 cm long bed of tabular alumina (3 to 4 mm diameter) with large alumina balls (3 to 4 cm diameter) at the top and bottom of the filter. The large alumina balls are included for the purpose of reducing inlet turbulence and also to prevent the tabular alumina from obstructing the exit nozzle orifice. A cone shaped steel push-pull rod is

used as an on/off valve to regulate melt flow through the filter.

The filter bed preparation consists of heating the SiC tube to 970-1070°K and adding to it approximately 13 Kg of molten P1020 aluminum at a temperature of 970°K. The alumina balls and tabular alumina were separately heated to a red-hot temperature and were then added to the melt contained in the SiC tube with frequent tamping to prevent channeling. In this manner the filter bed is infiltrated with aluminum prior to filtration and hence ensures wetting of the filter grains.

The melt to be filtered is prepared by melting about 70 Kg of P1020 aluminum and heating it to 1020°K in an oil fired furnace. TiB_2 inclusions were chosen as the tracer inclusion to monitor the filtration efficiency. "Synthetic inclusions" were intentionally formed in the melt by adding a predetermined amount of titanium diboride (Al-5% Ti-1% B master) to obtain an initial inclusion concentration (C_i) in the range of 500-1000 ppm. Titanium diboride "synthetic inclusions" were chosen for several reasons: (i) the size of TiB_2 particulates is in the critical size range required in this study, i.e., 1 to 30μm; (ii) ease of quantitative analysis of Ti and B in aluminum via spectrographic and metallographic techniques; (iii) availability of Al-Ti-B master alloys.

Under the exit orifice of the filter the filtered metal is collected using a series of molds. By collecting the metal over a specific period of time and determining its weight, we were able to determine the melt flow-rate through the filter.

A filtration run consists of first stirring the melt rigorously in order to prevent settling of the TiB_2 inclusions, also the melt is subsequently restirred on a systematic basis throughout the duration of the experiment. The melt is then poured into the top of the SiC tube containing the filter bed with particular care being taken to maintain a constant metallostatic

head. It has been found that with this method flowrate variations can be kept within 10% error. Once the initial P1020 infiltrant aluminum has been purged out of the filter and the contaminated melt is flowing through the bed, inlet and filtered outlet samples were obtained for subsequent analysis for TiB_2 content. It is assumed that at the filtration temperature all of the boron present is chemically bound to titanium.

To date all of the filtration runs have been performed using (3 to 4 mm) tabular alumina as the filter medium, however future work is planned using a different size range of alumina tabs and data is being gathered on other candidate filter media in order to assess their inertness, thermal shock resistance, erosion behavior, wetting properties, etc.

In tandem with the deep bed filtration program, we are evaluating the performance of open pore ceramic filters. These are rigid filters where the extent of porosity is in the order of 90%; when exhausted the whole filter can be removed and a new one inserted and thus the name cartridge type filters. The experimental procedure is identical to the deep bed program with the exception that the open pore ceramic filter is 5 cm thick compared to the 46 cm height of the deep bed alumina tab filter. Performance of these two types of filters will be compared and analyzed.

4.2 Experimental Design for Steel Filtration

Using the experience gained in the aluminum filtration, a design for a steel filtration apparatus has now been finalized and actual assembly of the apparatus is in progress. A schematic plan of the apparatus is shown in Figure 4. The apparatus consists of an outer pressure vessel which will be divided into two compartments. The upper half houses the alumina crucible which contains the filter bed and the melt (charge) to be filtered.

The crucible is heated by induction via susceptors. The filter bed consists of tabular alumina particles of 3 mm in diameter. The bed itself is 50 mm in diameter and its height can be varied up to 350 mm. To prevent the alumina particles from floating they are restrained by an alumina disk with orifices for the melt to flow through. At the underside, the filter bed is supported by another alumina disk with a predetermined size orifice which restricts and controls melt flowrate through the bed. The lower half of the vessel contains the mold which will subsequently receive the filtered melt. The weight of the crucible is monitored by a load cell so that one can accurately determine the instantaneous flowrate of the filtered melt. Both chambers of the apparatus may be evacuated or exposed to argon at similar or differing pressures, the values of which are recorded by pressure indicators connected to each chamber.

The proposed experimental procedure is as follows: The melt is prepared by charging 4 Kg of electrolytic iron with a known oxygen level into the crucible. The chambers are evacuated and a constant argon purge is introduced. The charge is then heated by induction until a melt temperature of 1870°K is attained; a predetermined amount of aluminum pellets is then charged into the crucible which quickly dissolves under the vigorous stirring effect of the electromagnetic field. The literature indicates that Al_2O_3 is the primary deoxidation product when the oxygen level of the melt is less than 0.058%, while at higher oxygen levels hercynite forms[21]. At the present, melts are being prepared with known amounts of aluminum and oxygen, and the deoxidation products are being categorized. This is a necessary step before filtration experiments with steel melts are carried out. Using this information, a melt with known inclusions can be prepared and caused to flow through the filter by an argon pressure differential of 20 to 40 psi applied between the two chambers. When all the melt flows through the filter the pressure between the two chambers is equalized indicating the end of the run.

Filtration performance is evaluated by analyzing inlet and outlet samples for soluble and insoluble aluminum and total oxygen content. Thanks to Dr. Harry Paxton of U. S. Steel Laboratories, the chemical analyses are being evaluated at U. S. Steel's laboratories. Methods of analyzing size distribution of filtered and unfiltered melt inclusions are being looked into to establish the size range of inclusions being removed.

4.3 Modeling via Low Temperature Systems

Low temperature model systems for the removal of solid and liquid inclusions are being screened for the study of the capture mechanisms and kinetics during depth filtration. With model systems where the fluid is at ambient temperature one can visually observe the filtration process by using plexiglass containers for the filter bed. In addition, samples of filtered fluid can be obtained at any length throughout the filter bed via syringe type ports along the plexiglass column.

A great deal of work has been done in filtration of aqueous systems, where electrokinetic forces are dominant. For melt systems electrokinetic forces are absent and thus the model system simulating melt systems must be such that these forces are absent. At present, a non-polar fluid such as diesel fuel has been chosen with $CaCO_3$ contaminants. Filtration studies similar to the work for melt systems will be carried out with the model system. This work is presently underway.

4.4 Results and Discussion - Aluminum Filtration

The data obtained from experiments to date along with calculated parameters discussed in section 2.2 is shown in Table I. These results are graphically represented in Figures 5 to 9.

Figure 5 shows the effect of melt flowrate on filtration performance. The reciprocal of the flowrate ($1/U_m$) is plotted as a function of a pseudo-efficiency term ($\ln C_i/Co$). It can be seen that for a given filter height, higher filtration efficiencies are obtained at low melt approach velocities.

This behavior is further reflected in Figure 6 which shows the fraction of inclusions removed (η) given by $(C_i-C_o)/C_i$ as a function of the melt approach velocity. Note that at low velocities the fraction of the inclusions removed approaches unity and at high velocities (about 0.7 cm/sec) it approaches 0.5 to 0.6. It is believed that at even higher velocities the fraction of inclusions removed will be finite. This variation in filtration efficiency with approach velocity is consistent with the greater interfacial fluid shear in the packed bed at high flowrates causing dislodgement of entrapped inclusions.

The effect of melt approach velocity, indicated by the particle Reynolds Number, Re_p, on the kinetic constant, K_o is shown in Figure 7. It can be noted that K_o has a tendency to increase with increasing melt velocity.

The parameter λ , the filtration coefficient (K_o/U_m) which is directly related to the removal efficiency, may also be plotted as a function of interstitial melt velocity in Figure 8, where it can be seen that it decreases steadily with increasing velocities. If this relationship is reinterpreted by taking the reciprocal of λ, denoted by Λ, the unit removal distance, and plotting the latter as a function of melt velocity on a log/log ordinate produces, as would be expected, a linear relationship (Figure 9). From this type of a plot it is possible to predict what filter length would be required to remove 62.3% of inclusions for any melt approach velocity. This would, of course, be of great importance industrially in predicting filter efficiencies for specific filter factors.

5. FUTURE WORK

Work is planned in several different areas. Filtration of aluminum and steels is progressing in tandem, and specifically the following tasks will be addressed:

Aluminum Filtration

- evaluate depth filters of different heights and various different filter grain size.
- determine the "useful life" of depth filters.
- evaluate the performance of open pore ceramic filters.

Steel Filtration

- categorize and identify inclusions in the model electrolytic iron melt.
- evaluate filtration efficiency using Al_2O_3 tabular beds.

In addition, work is planned utilizing the low temperature model system where the feasibility of capturing liquid inclusions will be evaluated and modeled. Filtration of superalloys will be undertaken after the above tasks are completed.

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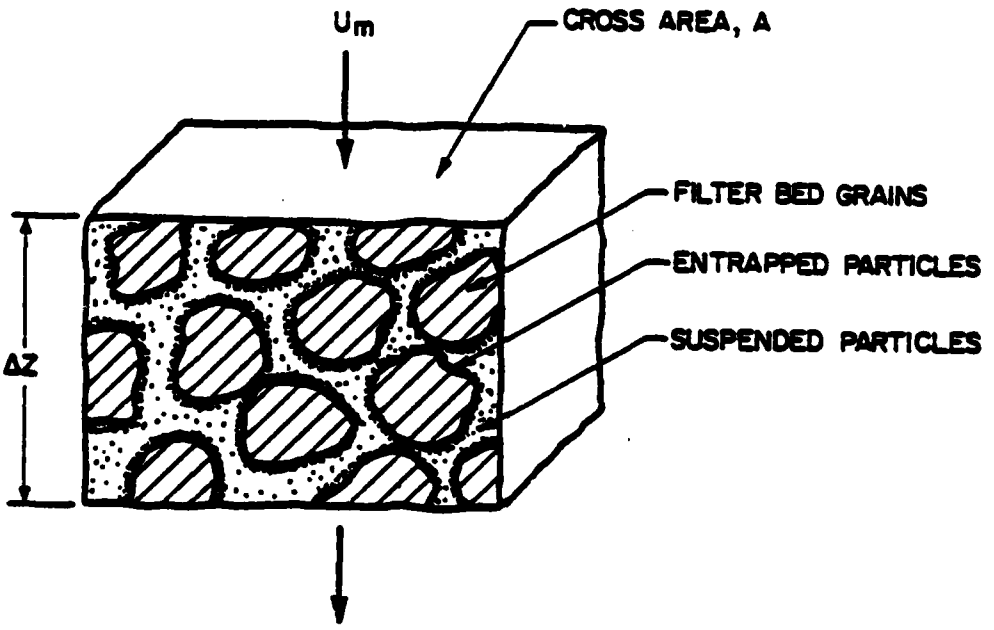


Figure 1. Differential Cross-section of Depth Filter

TABLE I

Aluminum Depth Filtration Results

L ins.	U_m cm/sec	C_i ppm.vol	C_o ppm.vol	η	λ^{-1} cm^{-1}	K_o^{-1} $sec.^{-1}$	Λ cm
20	0.3649	225	107	0.52	0.0146	0.0055	68.49
18	0.443	375	140	0.63	0.0217	0.0096	46.08
18	0.443	175	110	0.37	0.0101	0.0045	98.47
18	0.443	160	70	0.56	0.0181	0.0080	55.25
18	0.145	344	107	0.69	0.0256	0.0037	39.06
18	0.145	283	65	0.77	0.0323	0.0047	30.96
18	0.4431	250	80	0.68	0.0248	0.0110	40.32
18	0.4431	430	140	0.67	0.0244	0.0108	40.98
18	0.3145	710	210	0.70	0.0272	0.0085	36.76
18	0.3145	730	260	0.64	0.0224	0.0070	44.64
18	0.2439	650	200	0.69	0.0260	0.0063	38.46
18	0.1220	550	95	0.83	0.0386	0.0047	25.91
18	0.7328	736	268	0.64	0.0220	0.0161	45.45
18	0.7328	1120	520	0.54	0.0168	0.0037	59.52
18	0.0541	510	12	0.98	0.0819	0.0044	12.21
18	0.0541	842	153	0.98	0.0878	0.0048	11.39
18	0.0541	700	25	0.96	0.0728	0.0039	13.74
18	0.1839	555	42	0.92	0.0563	0.0104	17.76
18	0.1740	550	38	0.93	0.0583	0.0102	17.15
18	0.1740	525	59	0.89	0.0480	0.0083	20.83
18	0.1652	475	75	0.84	0.0406	0.0067	24.63
18	0.1652	435	62	0.86	0.0425	0.0070	23.53
18	0.1477	570	69	0.88	0.0461	0.0068	21.69
18	0.1160	470	57	0.88	0.0461	0.0054	21.69
18	0.0894	480	54	0.89	0.0476	0.0043	21.01
18	0.0819	560	53	0.90	0.0496	0.0041	20.16
6	0.0750	237	101	0.57	0.0560	0.0043	17.86
6	0.0750	223	105	0.53	0.0494	0.0038	20.24
6	0.0750	190	83	0.56	0.0543	0.0041	18.42
18	0.05	475	40	0.92	0.0539	0.0027	18.55
18	0.05	528	51	0.90	0.0512	0.0026	19.53

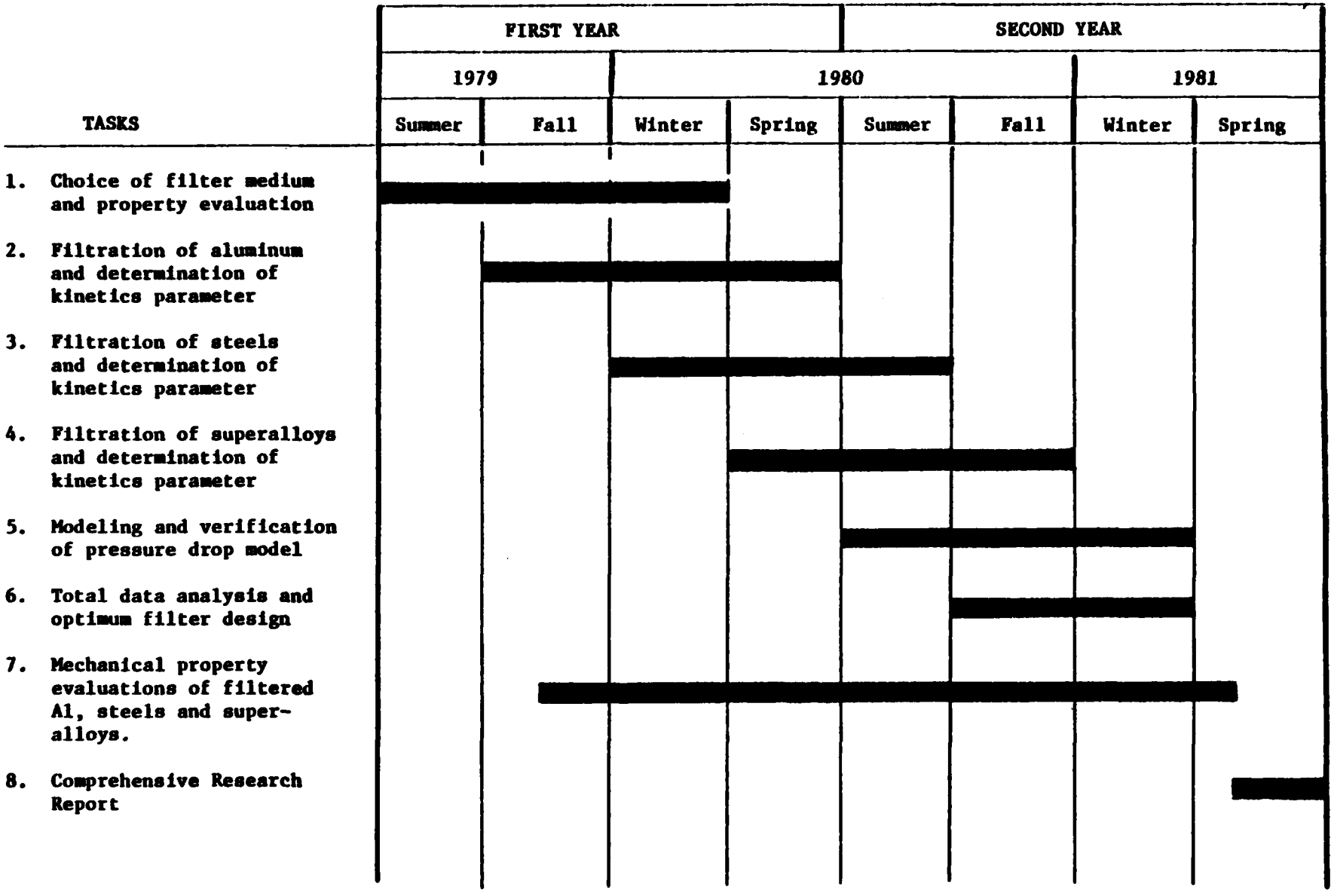


Figure 2. Proposed Program Schedule

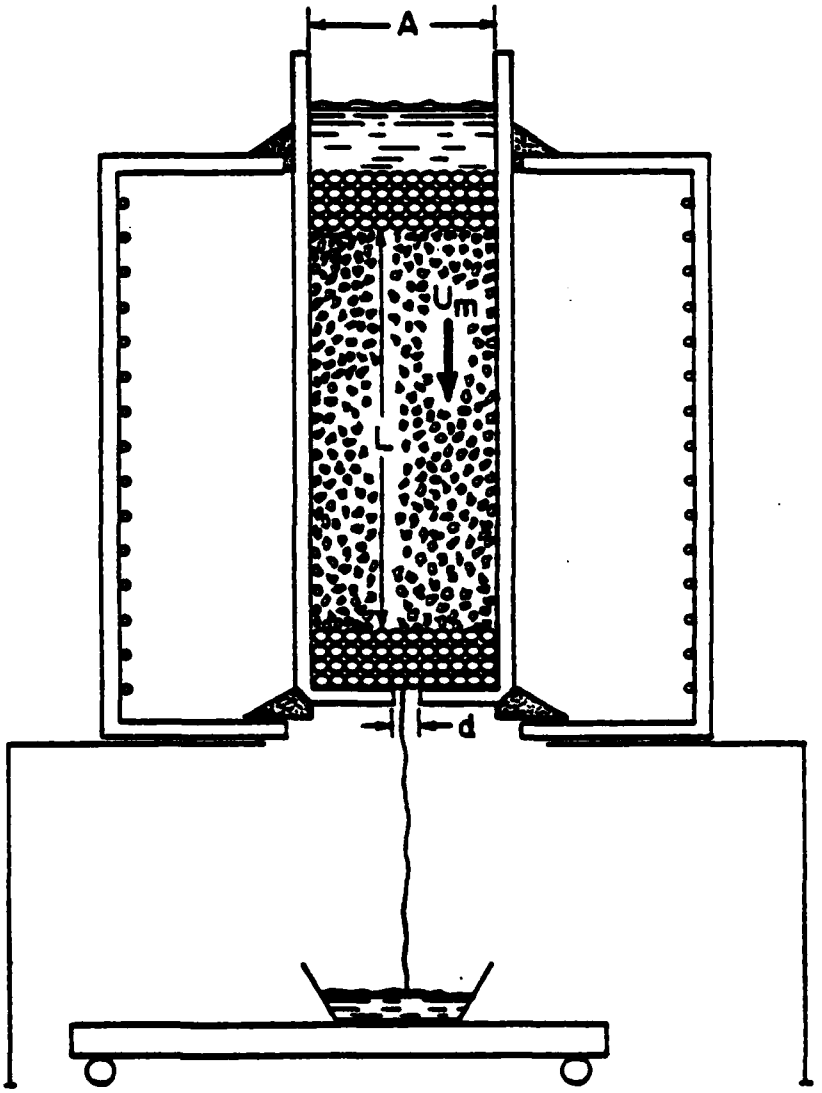
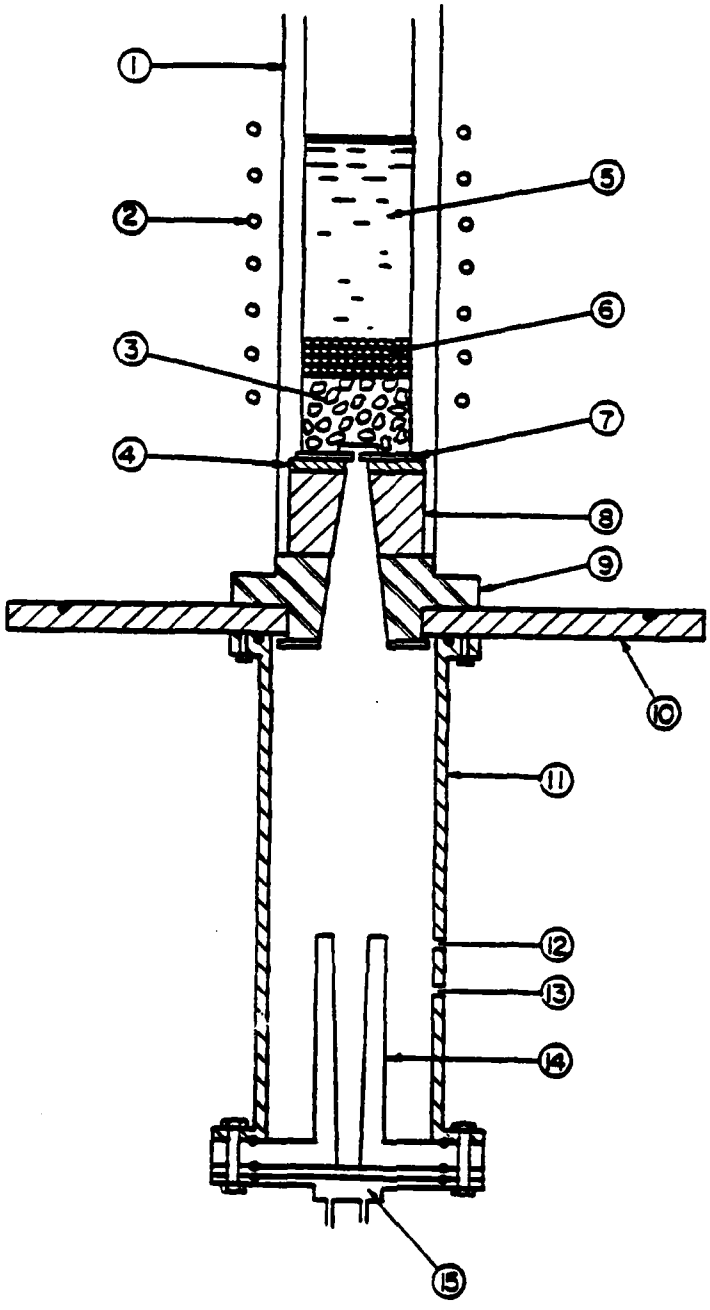


Figure 3. Schematic Diagram of Aluminum Filtration Apparatus.



Legend

1. Graphite susceptor
2. Induction coil
3. Filter bed
4. Al_2O_3 disc
5. Steel melt
6. Particle valve
7. Orifice disc
8. Pyrolitic graphite
9. Stainless steel pedestal
10. Base plate supporting dome
11. Lower chamber
12. Vacuum fittings
13. T/C fittings
14. Mold
15. Chill

Figure 4. Schematic diagram of Steel Filtration Apparatus

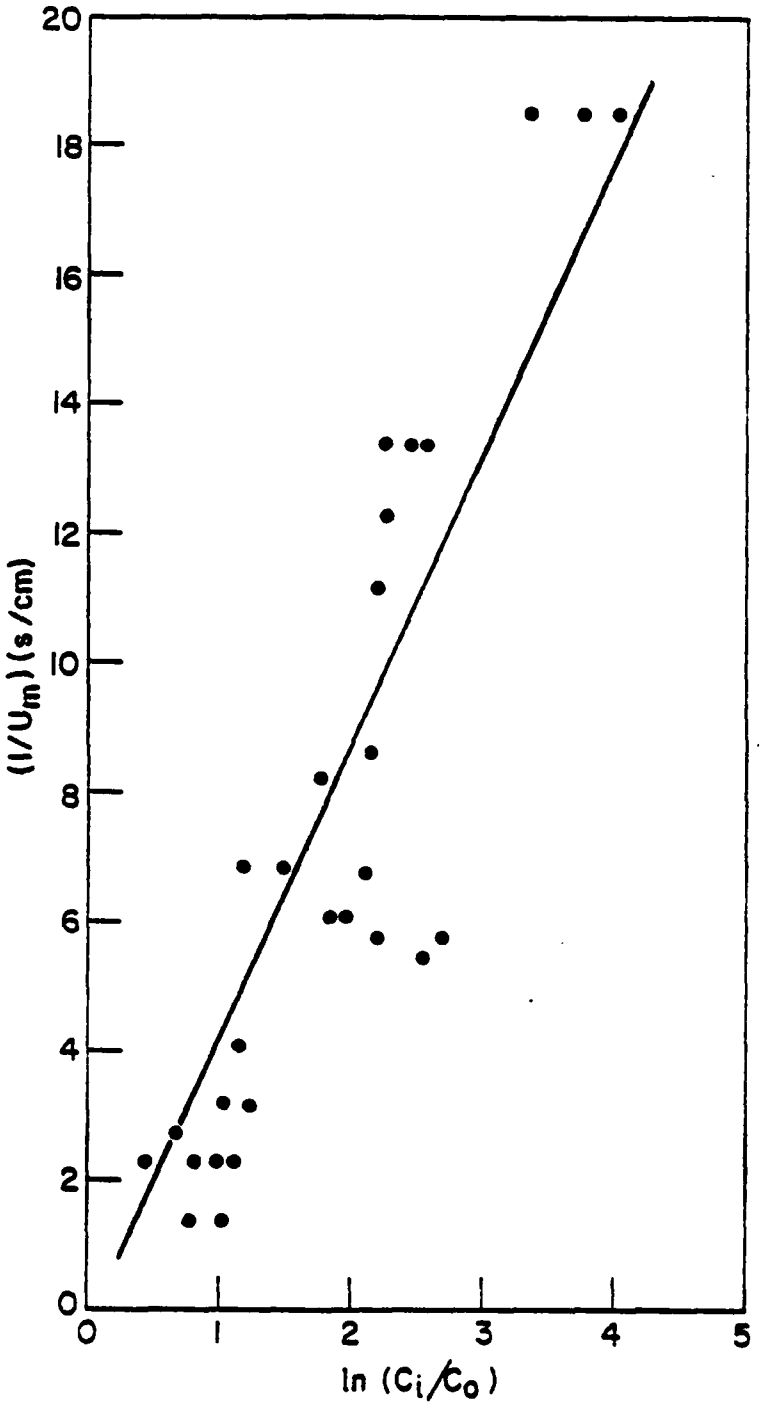


Figure 5. Effect of Superficial Melt Velocity on the function.

$M \frac{Ci}{Co}$

Slope of the line is the reciprocal of $(K_o L)$

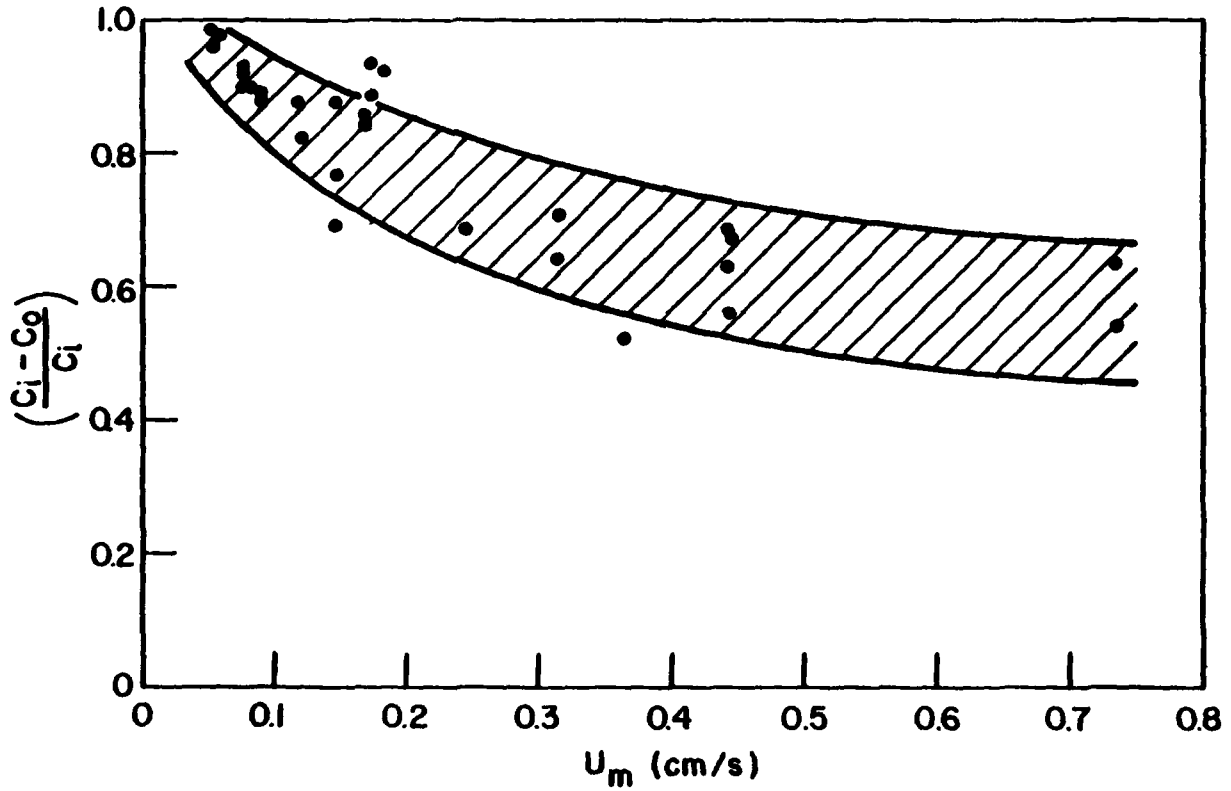


Figure 6. Effect of Superficial Melt Velocity on Filtration Efficiency.

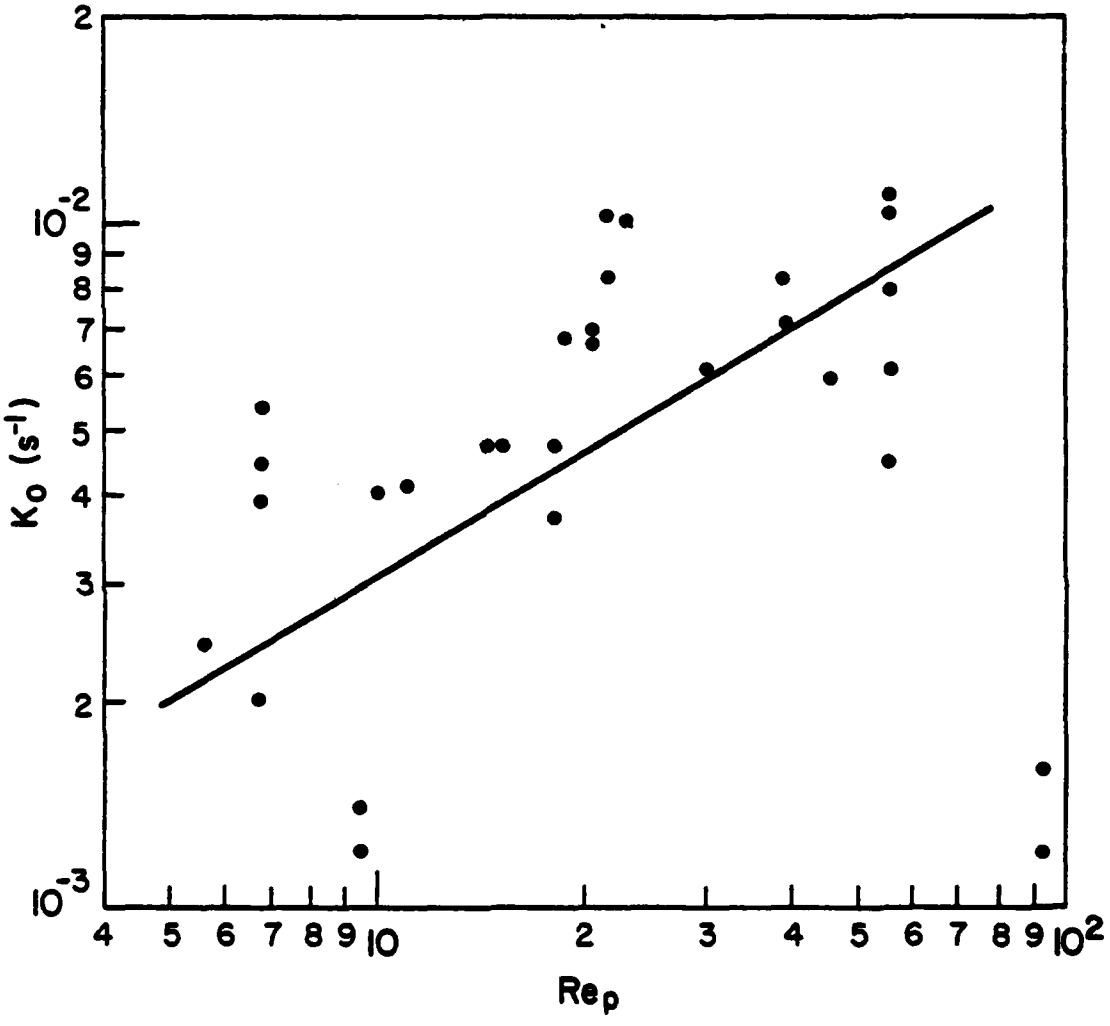


Figure 7. The Kinetic Parameter as a Function of Particle Reynolds Number.

Where: $Re_p = \frac{d_g U_m \rho}{\mu(1-\varepsilon)}$

d_g = particle diameter

Um = superficial melt velocity

ρ = melt density

μ = melt viscosity

ε = bed porosity

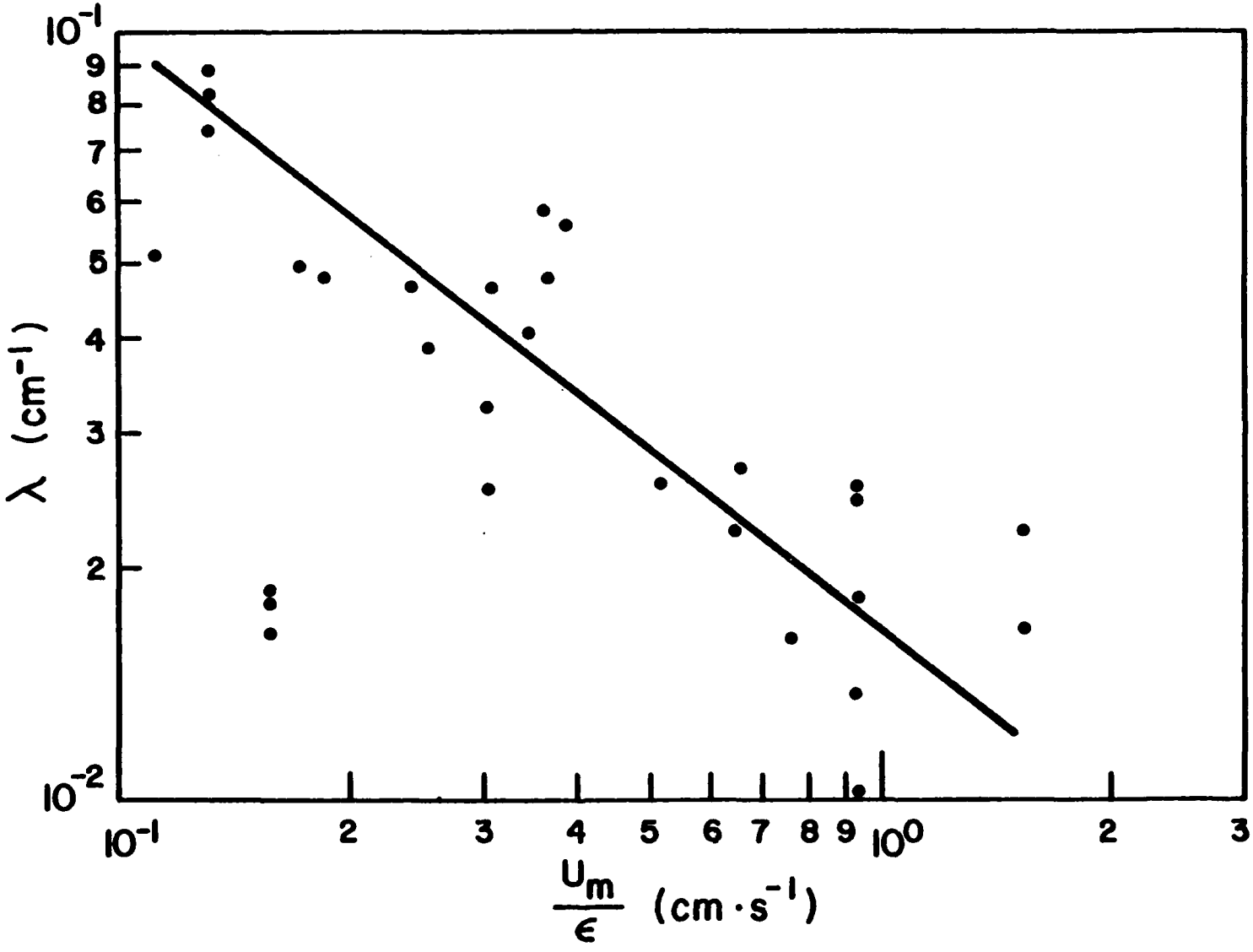


Figure 8. The Filtration Coefficient λ as a Function of Interstitial Velocity (where interstitial velocity = U_m/ϵ. The bed porosity, ϵ, was measured as 42%).

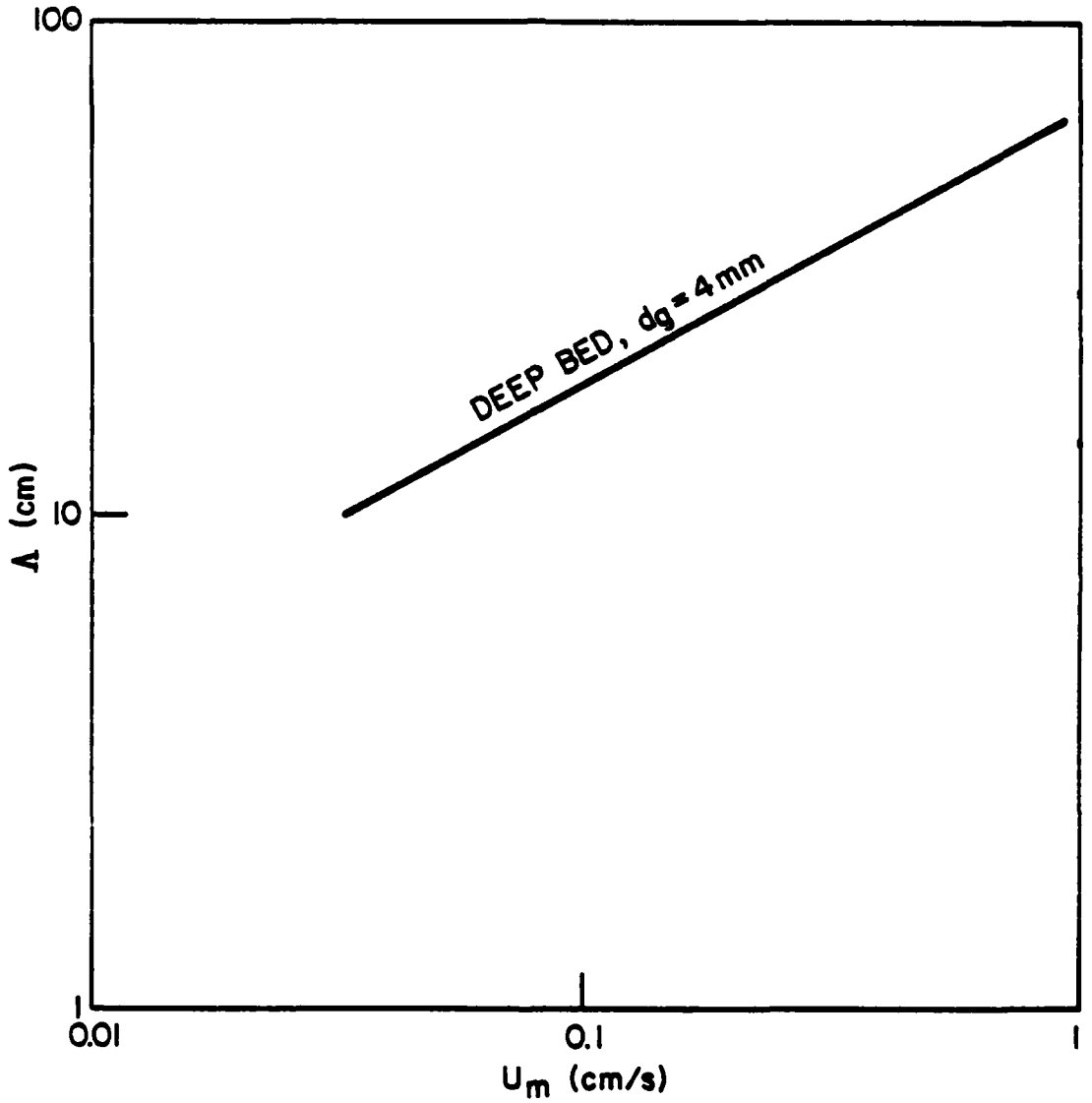


Figure 9. The Unit Removal Distance as a Function of Superficial Melt Velocity.

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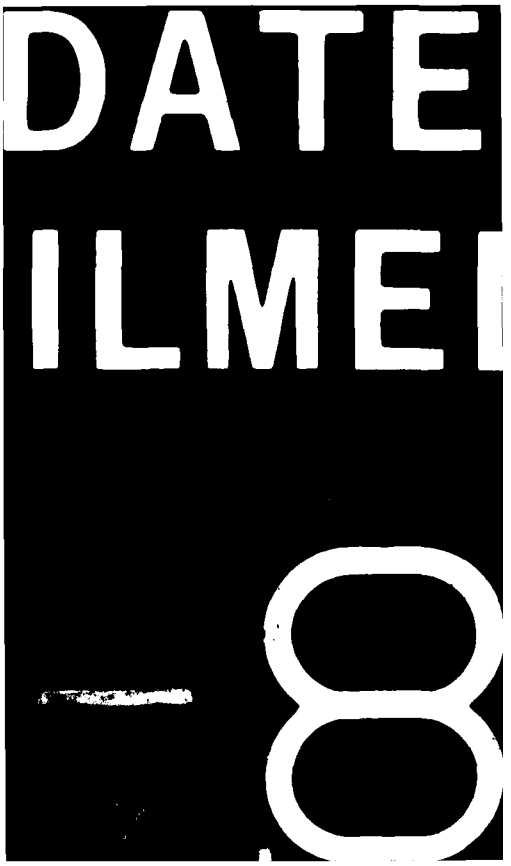
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